1976), F magnitudes, $\sigma^{-2}(F)$ weights, all non-H atoms anisotropic, all H atoms isotropic in calculated positions (C—H = 1·08 Å, H—C—H = 109·4°) with a common thermal parameter that was also refined [U_{iso}(H) = 0·138 (6) Å^2], 580 variables refined, $\sum w|\Delta F|^2$ minimized. Final $wR = 0{\cdot}060$, $R = 0{\cdot}083$, $(\Delta/\sigma)_{max} < 0{\cdot}1$, maximum residual electron density = 0·43 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1 gives the atom parameters, Table 2 the bond lengths and valence angles, and Fig. 1 shows the molecular structure and atomic numbering scheme drawn by *ORTEP* (Johnson, 1965).*

Related literature. In the TATM–EtOAc clathrate studied recently (van Rooyen & Roos, 1990) the interaction of the guest molecule is with two acetylthienyl substituents of each host molecule.

The authors wish to thank Dr O. Meth-Cohn for the title compound.

* Lists of structure factors, bond angles involving H atoms, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54396 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(carbodifluoro) and Tris(carbodifluoro) Derivatives of Zaluzanin D*

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Abstract. (1) (3a*S*,6*S*,6a*R*,8*S*,9*S*,9a*R*,9b*S*)-2′,2′,2″,-2″-Tetrafluoro-3-methylene-2-oxo-2,3,3a,4,5,6,6a,7,-8,9,9a,9b-dodecahydrodispiro[azuleno[4,5-*b*]furan-6,1′:9,1″-biscyclopropan]-8-yl acetate, $C_{19}H_{20}F_4O_4$, $M_r = 388{\cdot}4$, monoclinic, $P2_1$, $a = 9{\cdot}912$ (3), $b = 8{\cdot}415$ (2), $c = 11{\cdot}096$ (3) Å, $\beta = 99{\cdot}98$ (2)°, $V = 911{\cdot}5$ (4) Å^3, $Z = 2$, $D_x = 1{\cdot}41$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 10{\cdot}52$ cm^{-1}, $F(000) = 404$, $T = 293$ K, $R = 0{\cdot}064$, $wR = 0{\cdot}073$ for 1182 observed reflections [$F > 3\sigma(F)$]. (2) (3*S*,3a*S*,6*S*,6a*R*,8*S*,9*S*,9a*R*,9b*S*)-2′,2′,2″,2″,2‴,2‴-Hexafluoro-3-methylene-2-oxo-2,3,3a,4,5,6,6a,7,8,9,9a,9b-dodecahydrotrispiro[azuleno[4,5-*b*]furan-6,1′:6,1″:9,1‴-triscyclopropan]-8-yl acetate, $C_{20}H_{20}F_6O_4$, $M_r = 438$, monoclinic, $P2_1$, $a = 10{\cdot}348$ (3), $b = 8{\cdot}388$ (2), $c = 11{\cdot}093$ (3) Å, $\beta = 97{\cdot}64$ (2)°, $V = 954{\cdot}3$ (4) Å^3, $Z = 2$, $D_x = 1{\cdot}53$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 12{\cdot}34$ cm^{-1}, $F(000) = 452$, $T = 293$ K, $R = 0{\cdot}033$, $wR = 0{\cdot}037$ for 1264 observed reflections [$F > 3\sigma(F)$]. The chiral centers formed during the difluorocarbenation of zaluzanin D were determined relative to the 7*S* chiral center as has been found in zaluzanins and most of the guaianolides isolated so far. The asymmetry of difluorocyclopropane ring can be proposed as $\delta_1 + = [(\delta_2 -) + (\delta_3 -)]$ rather than [$\delta + = 2\delta -$] as described before [Allen (1980). *Acta Cryst.* B**36**, 81–96]. Molecular packing in the crystals is due entirely to van der Waals interactions.

Experimental. Title compounds were synthesized as previously described by Salazar & Díaz (1978).

(1). Colorless crystals 0·04 × 0·22 × 0·52 mm from acetone–isopropyl ether. Intensity data from Nicolet *P3/F* diffractometer, Ni-filtered Cu radiation. Lattice parameters from 25 centered reflections (8·11 < 2θ < 29·20°), 1326 measured intensities with 3 < 2θ < 110°, 1240 unique, $R_{int} = 0{\cdot}28$. 58 unobserved [$F <$

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Table 1. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($Å^2 \times 10^3$)

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}
(1)				
C(1)	8394 (6)	705	7446 (5)	52 (2)
C(2)	7270 (7)	395 (9)	8264 (6)	63 (2)
C(3)	6971 (6)	2044 (8)	8765 (5)	54 (2)
C(4)	7733 (6)	3208 (7)	8097 (5)	50 (2)
C(5)	8097 (5)	2404 (8)	6967 (4)	44 (2)
C(6)	6940 (5)	2498 (7)	5860 (4)	43 (2)
O(2)	6742 (4)	4167 (5)	5506 (3)	52 (1)
C(7)	7274 (5)	1673 (8)	4717 (5)	48 (2)
C(8)	6792 (7)	−46 (9)	4539 (5)	60 (2)
C(9)	7242 (7)	−1114 (8)	5672 (7)	65 (2)
C(10)	8526 (6)	−568 (7)	6526 (5)	54 (2)
C(11)	6654 (6)	2762 (8)	3705 (5)	53 (2)
C(12)	6465 (5)	4339 (8)	4278 (5)	52 (2)
O(3)	6116 (5)	5569 (7)	3814 (4)	69 (2)
C(13)	6242 (10)	2520 (13)	2530 (6)	99 (4)
C(14)	9658 (8)	−1849 (9)	6934 (7)	78 (3)
C(15)	7462 (7)	5012 (9)	8223 (6)	64 (2)
C(16)	8739 (7)	4329 (10)	8832 (6)	74 (3)
F(1)	8989 (5)	4200 (7)	10073 (3)	102 (2)
F(2)	9956 (4)	4591 (6)	8477 (4)	91 (2)
C(17)	9798 (7)	−633 (10)	6067 (7)	71 (3)
F(3)	9848 (6)	−997 (7)	4889 (5)	99 (2)
F(4)	10759 (4)	521 (7)	6394 (5)	98 (2)
O(1)	5545 (4)	2453 (6)	8444 (3)	55 (1)
C(18)	4679 (7)	1775 (9)	9073 (5)	58 (2)
O(4)	5037 (6)	750 (8)	9849 (5)	84 (2)
C(19)	3261 (7)	2383 (11)	8697 (6)	73 (3)
(2)				
C(1)	1582 (3)	1597	2514 (2)	42 (1)
C(2)	2651 (3)	1278 (4)	1678 (3)	56 (1)
C(3)	3001 (3)	2932 (4)	1218 (2)	47 (1)
C(4)	2245 (3)	4109 (4)	1907 (2)	44 (1)
C(5)	1915 (3)	3269 (4)	3036 (2)	40 (1)
C(6)	3034 (3)	3258 (4)	4092 (2)	41 (1)
C(7)	2712 (2)	2387 (4)	5236 (2)	39 (1)
C(8)	3109 (3)	636 (4)	5313 (3)	50 (1)
C(9)	2576 (3)	−386 (4)	4216 (3)	55 (1)
C(10)	1387 (3)	268 (4)	3392 (3)	48 (1)
C(11)	3372 (3)	3440 (4)	6234 (2)	42 (1)
C(12)	3609 (3)	5001 (4)	5688 (3)	47 (1)
C(13)	4426 (3)	2834 (5)	7300 (3)	56 (1)
C(14)	295 (4)	−959 (5)	2972 (4)	69 (1)
C(15)	2468 (3)	5911 (4)	1784 (3)	55 (1)
C(16)	1282 (3)	5161 (4)	1213 (3)	55 (1)
C(17)	3131 (3)	3320 (4)	7522 (2)	51 (1)
C(18)	123 (3)	231 (5)	3879 (3)	62 (1)
C(19)	5219 (3)	2709 (4)	870 (3)	48 (1)
C(20)	6563 (3)	3324 (5)	1274 (3)	57 (1)
O(1)	4366 (2)	3304 (3)	1571 (2)	49 (1)
O(2)	4901 (3)	1841 (4)	31 (2)	71 (1)
O(3)	3303 (2)	4903 (3)	4474 (2)	48 (1)
O(4)	4037 (2)	6208 (3)	6172 (2)	64 (1)
F(1)	1023 (2)	5061 (3)	−14 (2)	78 (1)
F(2)	128 (2)	5434 (3)	1637 (2)	71 (1)
F(3)	2948 (2)	4619 (3)	8184 (1)	70 (1)
F(4)	2282 (2)	2192 (3)	7785 (2)	60 (1)
F(5)	−740 (2)	1441 (3)	3620 (2)	78 (1)
F(6)	80 (3)	−185 (3)	5043 (2)	87 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

(1)			
C(1)—C(2)	1·576 (10)	C(1)—C(5)	1·537 (7)
C(1)—C(10)	1·501 (7)	C(2)—C(3)	1·542 (10)
C(3)—C(4)	1·508 (9)	C(3)—O(1)	1·439 (7)
C(4)—C(5)	1·523 (8)	C(4)—C(15)	1·552 (10)
C(4)—C(16)	1·506 (9)	C(5)—C(6)	1·531 (6)
C(6)—O(2)	1·463 (8)	C(6)—C(7)	1·531 (8)
O(2)—C(12)	1·351 (7)	C(7)—C(8)	1·526 (10)
C(7)—C(11)	1·496 (8)	C(8)—C(9)	1·547 (9)
C(9)—C(10)	1·521 (8)	C(10)—C(14)	1·565 (10)
C(10)—C(17)	1·442 (10)	C(11)—C(12)	1·497 (10)
C(11)—C(13)	1·313 (9)	C(12)—O(3)	1·181 (9)
C(15)—C(16)	1·447 (10)	C(14)—C(17)	1·428 (11)
C(16)—F(1)	1·361 (7)	C(16)—F(2)	1·351 (9)
C(17)—F(3)	1·351 (9)	C(17)—F(4)	1·365 (9)
O(1)—C(18)	1·326 (8)	C(18)—O(4)	1·227 (9)
C(18)—C(19)	1·487 (9)		

Table 2 (*cont.*)

(2)			
C(1)—C(2)	1·558 (4)	C(1)—C(5)	1·538 (4)
C(1)—C(10)	1·512 (4)	C(2)—C(3)	1·538 (5)
C(3)—C(4)	1·526 (4)	C(3)—O(1)	1·448 (3)
C(4)—C(5)	1·515 (4)	C(4)—C(15)	1·538 (5)
C(4)—C(16)	1·469 (4)	C(5)—C(6)	1·534 (3)
C(6)—C(7)	1·539 (4)	C(6)—O(3)	1·459 (4)
C(7)—C(8)	1·525 (4)	C(7)—C(11)	1·507 (4)
C(8)—C(9)	1·530 (4)	C(9)—C(10)	1·533 (4)
C(10)—C(14)	1·554 (5)	C(10)—C(18)	1·480 (5)
C(11)—C(12)	1·477 (5)	C(11)—C(13)	1·582 (4)
C(11)—C(17)	1·486 (4)	C(12)—O(3)	1·344 (3)
C(12)—O(4)	1·203 (4)	C(13)—C(17)	1·452 (5)
C(15)—C(16)	1·448 (4)	C(14)—C(18)	1·445 (5)
C(16)—F(1)	1·354 (3)	C(16)—F(2)	1·360 (4)
C(17)—F(3)	1·342 (4)	C(17)—F(4)	1·349 (4)
C(18)—F(5)	1·357 (4)	C(18)—F(6)	1·344 (4)
C(19)—C(20)	1·495 (4)	C(19)—O(1)	1·348 (4)
C(19)—O(2)	1·193 (4)		

(1)			
C(2)—C(1)—C(5)	104·0 (4)	C(2)—C(1)—C(10)	115·3 (4)
C(5)—C(1)—C(10)	117·6 (5)	C(1)—C(2)—C(3)	105·0 (5)
C(2)—C(3)—C(4)	105·2 (5)	C(2)—C(3)—O(1)	111·6 (5)
C(4)—C(3)—O(1)	106·1 (5)	C(3)—C(4)—C(5)	109·1 (5)
C(3)—C(4)—C(15)	118·8 (6)	C(5)—C(4)—C(15)	125·3 (5)
C(3)—C(4)—C(16)	118·8 (5)	C(5)—C(4)—C(16)	119·7 (5)
C(15)—C(4)—C(16)	56·4 (4)	C(1)—C(5)—C(4)	100·8 (4)
C(1)—C(5)—C(6)	113·4 (5)	C(4)—C(5)—C(6)	112·7 (4)
C(5)—C(6)—O(2)	108·2 (4)	C(5)—C(6)—C(7)	113·7 (4)
O(2)—C(6)—C(7)	104·6 (4)	C(6)—O(2)—C(12)	111·6 (4)
C(6)—C(7)—C(8)	115·4 (5)	C(6)—C(7)—C(11)	103·0 (5)
C(8)—C(7)—C(11)	113·9 (5)	C(7)—C(8)—C(9)	114·0 (5)
C(8)—C(9)—C(10)	115·0 (6)	C(1)—C(10)—C(9)	118·5 (5)
C(1)—C(10)—C(14)	115·9 (5)	C(9)—C(10)—C(14)	117·0 (6)
C(1)—C(10)—C(17)	116·9 (5)	C(9)—C(10)—C(17)	117·3 (6)
C(14)—C(10)—C(17)	56·5 (5)	C(7)—C(11)—C(12)	106·8 (5)
C(7)—C(11)—C(13)	131·8 (7)	C(12)—C(11)—C(13)	121·2 (7)
O(2)—C(12)—C(11)	108·3 (5)	O(2)—C(12)—O(3)	121·8 (6)
C(11)—C(12)—O(3)	129·9 (5)	C(4)—C(15)—C(16)	60·2 (5)
C(10)—C(14)—C(17)	57·4 (5)	C(4)—C(16)—C(15)	63·4 (4)
C(4)—C(16)—F(1)	119·1 (7)	C(15)—C(16)—F(1)	119·7 (7)
C(4)—C(16)—F(2)	119·1 (6)	C(15)—C(16)—F(2)	123·5 (6)
F(1)—C(16)—F(2)	107·0 (5)	C(10)—C(17)—C(14)	66·1 (5)
C(10)—C(17)—F(3)	122·1 (6)	C(14)—C(17)—F(3)	120·9 (7)
C(10)—C(17)—F(4)	119·5 (6)	C(14)—C(17)—F(4)	118·4 (6)
F(3)—C(17)—F(4)	106·1 (6)	C(3)—O(1)—C(18)	117·7 (5)
O(1)—C(18)—O(4)	122·5 (6)	O(1)—C(18)—C(19)	111·8 (6)
O(4)—C(18)—C(19)	125·7 (7)		
(2)			
C(2)—C(1)—C(5)	103·9 (2)	C(2)—C(1)—C(10)	115·1 (3)
C(5)—C(1)—C(10)	118·1 (2)	C(1)—C(2)—C(3)	105·0 (3)
C(2)—C(3)—C(4)	105·0 (2)	C(2)—C(3)—O(1)	111·6 (2)
C(4)—C(3)—O(1)	105·8 (2)	C(3)—C(4)—C(5)	107·5 (3)
C(3)—C(4)—C(15)	119·9 (3)	C(5)—C(4)—C(15)	125·7 (3)
C(3)—C(4)—C(16)	118·9 (2)	C(5)—C(4)—C(16)	120·0 (2)
C(15)—C(4)—C(16)	57·5 (2)	C(1)—C(5)—C(4)	100·2 (2)
C(1)—C(5)—C(6)	113·0 (2)	C(4)—C(5)—C(6)	113·8 (2)
C(5)—C(6)—C(7)	114·1 (2)	C(5)—C(6)—O(3)	108·2 (2)
C(7)—C(6)—O(3)	105·2 (2)	C(6)—C(7)—C(8)	114·6 (2)
C(6)—C(7)—C(11)	101·6 (2)	C(8)—C(7)—C(11)	115·5 (2)
C(7)—C(8)—C(9)	115·4 (2)	C(8)—C(9)—C(10)	116·7 (3)
C(1)—C(10)—C(9)	118·8 (2)	C(1)—C(10)—C(14)	116·7 (2)
C(9)—C(10)—C(14)	115·9 (3)	C(1)—C(10)—C(18)	116·8 (3)
C(9)—C(10)—C(18)	117·0 (3)	C(14)—C(10)—C(18)	56·8 (2)
C(7)—C(11)—C(12)	107·6 (2)	C(7)—C(11)—C(13)	124·1 (3)
C(12)—C(11)—C(13)	117·1 (2)	C(7)—C(11)—C(17)	123·3 (3)
C(12)—C(11)—C(17)	120·8 (3)	C(13)—C(11)—C(17)	56·4 (2)
C(11)—C(12)—O(3)	109·1 (3)	C(11)—C(12)—O(4)	129·4 (3)
O(3)—C(12)—O(4)	121·5 (3)	C(11)—C(13)—C(17)	58·5 (2)
C(4)—C(15)—C(16)	58·9 (2)	C(10)—C(14)—C(18)	59·0 (2)
C(4)—C(16)—C(15)	63·6 (2)	C(4)—C(16)—F(1)	121·3 (3)
C(15)—C(16)—F(1)	120·6 (3)	C(4)—C(16)—F(2)	119·1 (3)
C(15)—C(16)—F(2)	120·5 (3)	F(1)—C(16)—F(2)	107·0 (2)
C(11)—C(17)—C(13)	65·1 (2)	C(11)—C(17)—F(3)	121·7 (3)
C(13)—C(17)—F(3)	121·4 (3)	C(11)—C(17)—F(4)	116·9 (2)
C(13)—C(17)—F(4)	118·8 (3)	F(3)—C(17)—F(4)	107·9 (2)
C(10)—C(18)—C(14)	64·1 (2)	C(10)—C(18)—F(5)	119·7 (3)
C(14)—C(18)—F(5)	120·5 (3)	C(10)—C(18)—F(6)	120·0 (3)
C(14)—C(18)—F(6)	120·8 (3)	F(5)—C(18)—F(6)	107·1 (3)
C(20)—C(19)—O(1)	110·8 (3)	C(20)—C(19)—O(2)	126·4 (3)
O(1)—C(19)—O(2)	122·8 (3)	C(3)—O(1)—C(19)	117·0 (2)
C(6)—O(3)—C(12)	111·1 (2)		

Table 3. *Asymmetry of the cyclopropane ring (distances in* Å)

Cyclopropane* ring	S	s	R_1R_2	R_3R_4	R_5R_6	R	σ	D_1	D_2	D_3	Δ	δ_1	δ_2	δ_3	D_4
(1)															
C(16)—C(14)—C(4)	200	202	F,F	H,H	Cr,Cr	7·3	10	1·552	1·506	1·447	1·502	+50	−0·4	−55	1·356
C(17)—C(15)—C(10)	200	202	F,F	H,H	Cr,Cr	7·3	10	1·565	1·442	1·428	1·478	+87	−36	−50	1·358
(2)															
C(16)—C(14)—C(4)	200	202	F,F	H,H	Cr,Cr	3·3	4	1·538	1·469	1·448	1·485	+53	−16	−37	1·357
C(17)—C(13)—C(11)	200	202	F,F	H,H	Cr,Cr	3·3	4	1·582	1·486	1·452	1·507	+75	−21	−55	1·346
C(18)—C(15)—C(10)	200	202	F,F	H,H	Cr,Cr	3·3	5	1·554	1·480	1·445	1·493	+61	−13	−48	1·351
All	200	202	F,F	H,H	Cr,Cr	—	7	1·558	1·477	1·444	1·493	+85	−16	+49	1·353

* Column headings have the same meaning as in Allen (1980).

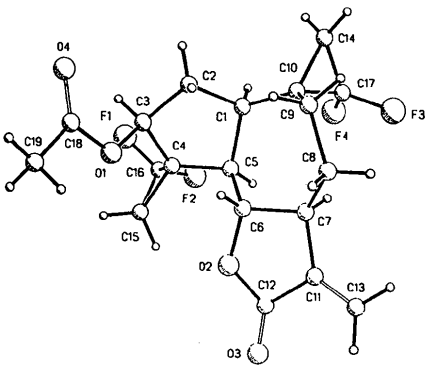

Fig. 1. Molecular structure of (1)

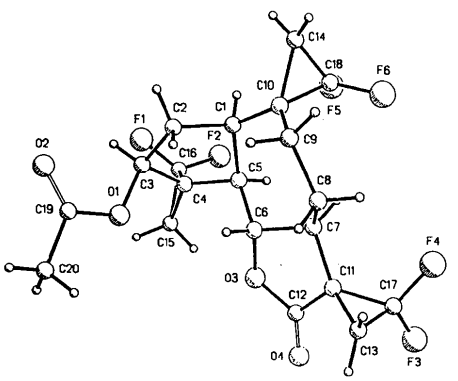

Fig. 2. Molecular structure of (2)

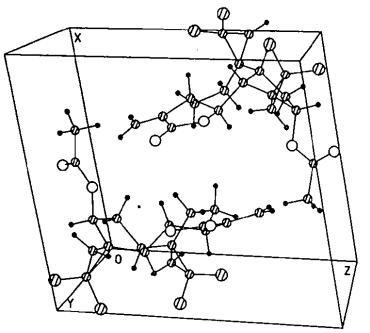

Fig. 3. Crystal packing of (1).

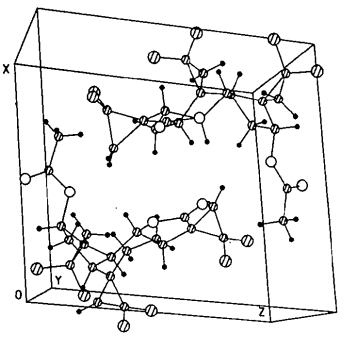

Fig. 4. Crystal packing of (2).

$3\sigma(F)$], index ranges: h 0 to 10; k 0 to 8; $l \pm 11$. $\theta/2\theta$ scan mode, scan range $[2\theta(K\alpha_1) - 1{\cdot}1]$ to $[2\theta(K\alpha_2) + 1{\cdot}1]°$, variable scan speed (min. 4·0, max. 29·3° min^{-1}), two standards (011, 121), decay ±2%, Lp corrections applied, absorption ignored, space group from Laue symmetry and systematic absences: $0k0$ for k odd. Structure solved by direct methods using *SHELXTL* (Sheldrick, 1983). Block-matrix least-squares refinement of 244 parameters based on F, including positional and anisotropic temperature parameters for non-H atoms, a scale factor and an isotropic extinction parameter. H atoms in idealized positions with fixed isotropic temperature parameter $U = 0{\cdot}06$ Å^2. Function minimized, $\sum w(\Delta F)^2$, $w = [\sigma^2(F_o) + 0{\cdot}004(F_o)^2]^{-1}$, origin by fixing the y coordinate of F(1). In last cycle $(\Delta/\sigma)_{max} = 0{\cdot}039$, $\Delta\rho$ −0·20 and 0·53 e Å^{-3}, $R = 0{\cdot}064$, $wR = 0{\cdot}073$, $S = 1{\cdot}44$, secondary-extinction correction parameter, $x = 0{\cdot}010$ (4) $[F_c = F_c/(1{\cdot}0 + 0{\cdot}02xF_c^2)^{0{\cdot}25}]$.

Compound (2) as for (1), but with specific values described below. Colorless crystals 0·10 × 0·18 × 0·50 mm from acetone–isopropyl ether. 1299 unique, $R_{int} = 0{\cdot}01$, 35 unobserved $[F < 3\sigma(F)]$. Scan range $[2\theta(K\alpha_1) - 1{\cdot}0]$ to $[2\theta(K\alpha_2) + 1{\cdot}0]°$, two standards (301, 201), decay ±2%. $w = [\sigma^2(F_o) + 0{\cdot}003(F_o)^2]^{-1}$, $(\Delta/\sigma)_{max} = 0{\cdot}19$, $-0{\cdot}15 \le \Delta\rho \le 0{\cdot}19$ e Å^{-3}, $R = 0{\cdot}033$, $wR = 0{\cdot}037$, $S = 0{\cdot}93$, $x = 0{\cdot}013$ (2), 272 parameters. All computations using complex atomic scattering factors from *International Tables for X-ray*

Crystallography (1974, Vol. IV) on a NOVA 4S computer.

Final atomic coordinates are given in Table 1.* Bond lengths and angles are shown in Table 2. Asymmetry parameters of the cyclopropane ring are given in Table 3. Ball and stick drawings of the molecules with the numbering schemes are shown in Figs. 1 and 2. Crystal packing is displayed in Figs. 3 and 4.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54388 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The structures of (1) and (2) have been previously deduced by NMR studies (Díaz, Ontiveros, Salazar, Negrón & Joseph-Nathan, 1981). Gas-phase studies on 1,1-difluorocyclopropane (Perreta & Laurie, 1975).

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Bis(carbodifluoro) Derivative of Anhydroparthenin*

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Abstract. (3*S*,6*S*,6a*S*,9a*R*)-6,6a-Difluoromethano-2′,2′-difluoro-6,7-dimethyl-2,3,3a,4,5,6,6a,7,8,9,9a,-9b-dodecahydrospiro[azuleno[4,5-*b*]furan-3,1′-cyclopropane]-2,9-dione, $C_{17}H_{16}F_4O_3$, $M_r = 344{\cdot}31$, monoclinic, *C*2, $a = 19{\cdot}271\ (7)$, $b = 6{\cdot}430\ (2)$, $c = 12{\cdot}456\ (4)$ Å, $\beta = 91{\cdot}70\ (3)°$, $V = 1542{\cdot}6\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}48$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 11{\cdot}23$ cm^{-1}, $F(000) = 712$, $T = 296$ K, $R = 0{\cdot}046$ for 1020 observed reflections [$F_o > 3\sigma(F_o)$]. The X-ray analysis enabled us to correct the configuration of the difluorocyclopropane attached to C(1), C(10) previously assigned on the basis of NMR studies. The configuration is 1*S*,5*R*,10*S*,11*S* relative to 7*S* as has been found in most of the pseudoguaianolides isolated so far. Cyclopentenone, cycloheptane and γ-lactone rings adopt envelope, twist-boat and half-chair conformations, respectively. Average bond length asymmetry in cyclopropane: distal bond 1·570 (6), vicinal bonds 1·474 (5) and 1·441 (6) Å. Packing entirely due to van der Waals forces.

Experimental. Title compound was crystallized from an acetone–isopropyl ether solution and yielded light-yellow crystals. Crystal used for data collection 0·12 × 0·24 × 0·28 mm, Nicolet *P*3/*F* diffractometer with Ni-filtered Cu radiation. Lattice parameters from 25 machine-centered reflections with $9{\cdot}18 < 2\theta < 28{\cdot}68°$; 1074 reflections with $3 < 2\theta < 110°$ were measured, 1051 unique, $R_{int} = 0{\cdot}042$, 23 unobserved [$F_o < 3\sigma(F_o)$], index ranges: *h* 0 to 20; *k* 0 to 6; *l* ±13. $\theta/2\theta$ scan mode, scan range [$2\theta(K\alpha_1) - 1{\cdot}0$] to [$2\theta(K\alpha_2) + 1{\cdot}0$]°, variable scan speed (min. 2·0, max. 29·3° min^{-1}), two standards (202, 312) monitored every 50 measurements, decay 3%, Lp corrections, absorption corrections ignored. Space group from systematic absences: *hkl* with $h + k$ odd, *C*2 or *C*2/*m*; successful structure solution and refinement proved *C*2 to be correct. Structure solved by direct methods using *SHELXTL* (Sheldrick, 1983), origin definition by fixing the *y* coordinate of F(1). Block-matrix least-squares refinement of 217 parameters based on *F*, including positional and anisotropic temperature parameters for non-H atoms and a scale factor. H atoms in idealized positions with fixed $U = 0{\cdot}06$ Å^2. Function minimized $\Sigma w(\Delta F)^2$; $w = [\sigma^2(F_o) + 0{\cdot}043(F_o)^2]^{-1}$. In last cycle $(\Delta/\sigma)_{max} = 0{\cdot}52$, residual electron density −0·26 and 0·21 e Å^{-3}. Final $R = 0{\cdot}046$, $wR = 0{\cdot}051$ and $S = 1{\cdot}06$, no extinction correction. Complex atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). All computations performed on a Data General NOVA 4S computer with the *SHELXTL* program package.

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